Consolidation of Advanced Powders by Severe Plastic Deformation

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Collaborators

- I. Anderson, DOE Ames Laboratory and Iowa State University, (Powder fabrication)
- R. Barber, Texas A&M University, (ECAE processing)
- J.T. Im, Texas A&M University, (Amorphous materials characterizations)
- H.J. Maier, University of Paderborn, (Transmission electron microscopy)
- J. Robertson, Texas A&M University, (Amorphous materials characterizations)

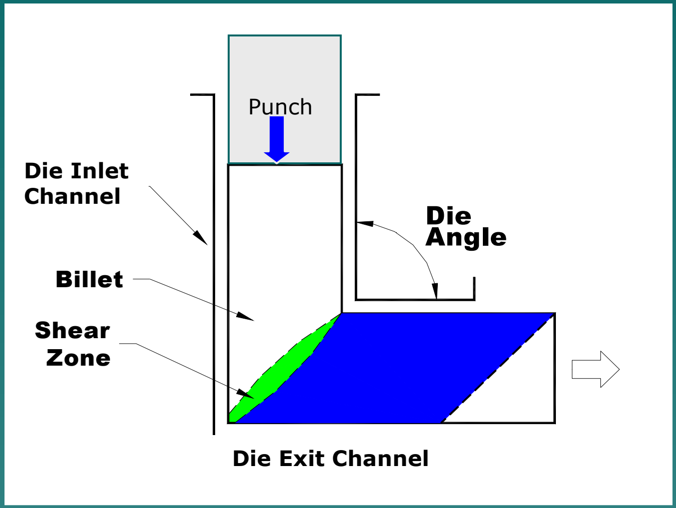
NATO-ARW

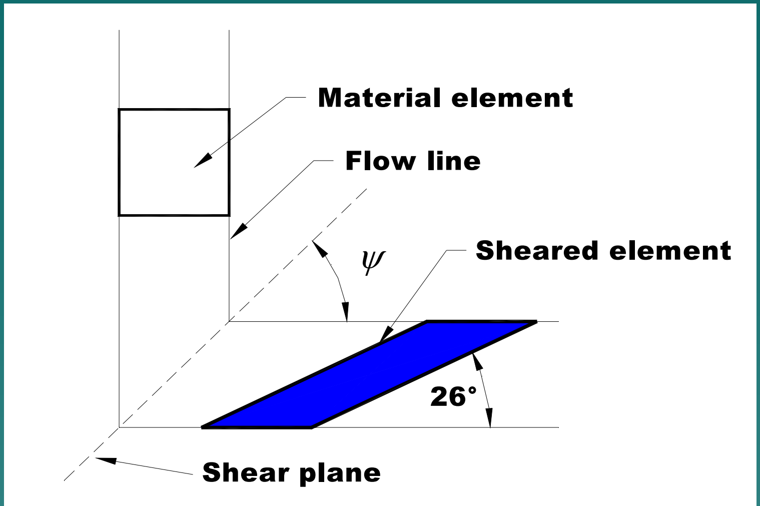
“Consolidation of Advanced Powders by Severe Plastic Deformation“

Talk Outline

1. Description of ECAE
2. Materials
   - Bulk Nanostructured Cu
   - Bulk Amorphous Zr-based Alloy
3. Experimental Results
4. Lessons Learned
5. Questions Remain

ECAE Concept





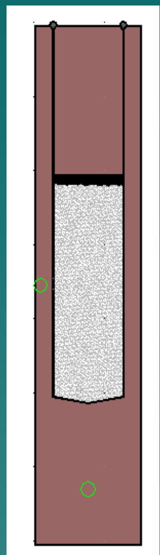
Conditions

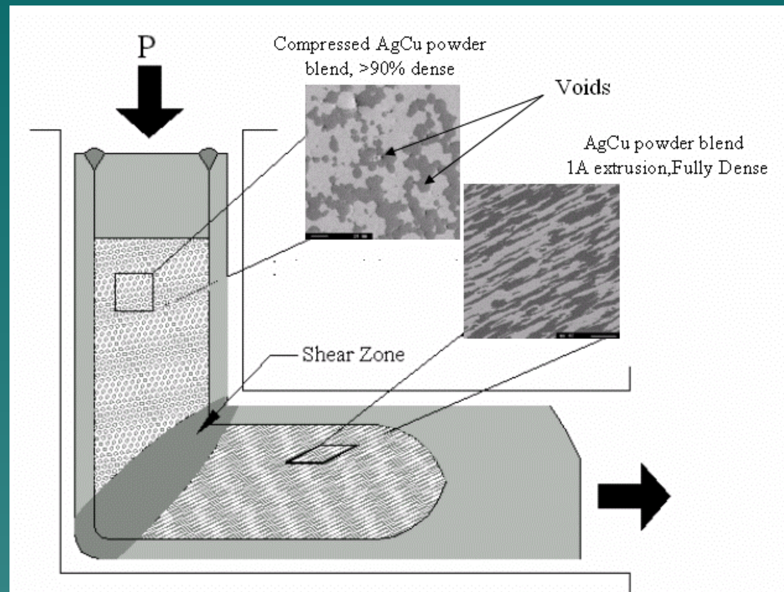
1. Inlet and outlet channels have nearly the same dimensions
2. Channel intersection is abrupt
3. Lubrication and other means are used to reduce friction

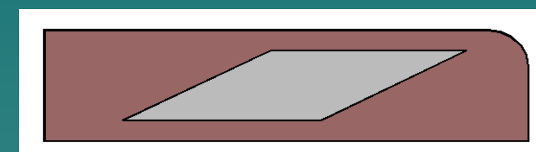
Results

1. Simple shear occurs
2. Effective strain is $(2/\sqrt{3}) \cot \psi$ or 1.16 for $\psi=45°$
3. Effective strain for multiple (N) extrusions is 1.16 N for $\psi=45°$
4. Strain is relatively uniform

Consolidation of powder by ECAE







Can/Powder Description

- **Inert Can Material**
- **0.75 x 0.75 x 3.5 inch**
- **0.50∅ x 1.5 inch Long Cavity**
- **Loose Powder with ~0.35 Void Fraction**
- **Vacuum Bake/Outgas**
- **e-beam Weld Seal**
- **Instrumented with Thermocouples**

Deformation Conditions

- **90^{o} Die Angle**
- **Isothermal Tool**
- **Constant Punch Speed**
- **Hydrostatic Pressure**
- **Simple Shear Uniformly Deforms Can and Encapsulate**
- **Heat of Deformation**
- **Collect Measurements**
  - **Load-Stroke**
  - **Time-Temperature**

Extruded Billet Characteristics

- **Near Full Density**
- **Shorter Billet (Cavity Length Decreases by ~1/3)**
- **Cavity Geometry Changes Shape (Depends on Number of Passes and Route)**

Potential Benefits of Powder Consolidation by ECAE

- Small heated cross-section relative to conventional area reduction extrusion (better heat transfer conditions)
- Large product cross-sections may be possible (conservation of cross-section during extrusion)
- High length/diameter ratio product may be possible
- Combined compaction and shear
- Consolidation to near full density after a single extrusion
- Consolidation to full density at lower temperature than needed for HIPing
- Lower punch loads than for area reduction extrusion

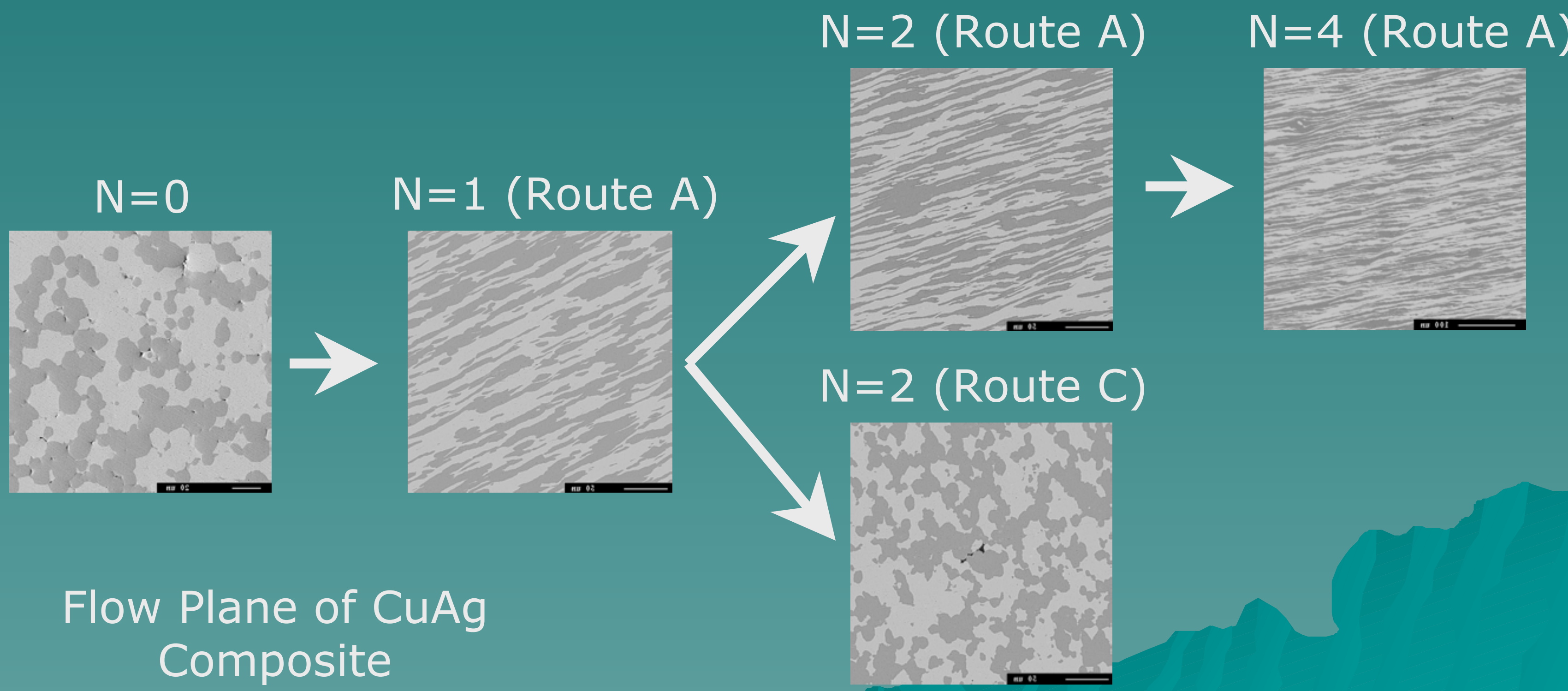
ECAE Route Descriptions

Route name	Min. # of passes	Billet rotations about the extrusion axis				Material Yield*	Effect on microstructure
		1 ➔	2 ➔	3 ➔	4 ➔ N		
A	1	0°	0°	0°	etc.	0.58	elongation (lamellar)
B (B_A)	2	+90°	-90°	+90°	etc.	0.67	elongation (filamentary)
C	2	180°	180°	180°	etc.	0.83	back/forth shearing
C' (B_C)	4	+90°	+90°	+90°	etc.	0.67	back/forth cross-shearing
E	4	180°	90°	180°	etc.	0.78	back/forth cross-shearing

* Theoretical yield of fully deformed material after N=4 in billet with length/width ratio of 6

Theoretical Change in Particle Surface Area for Different ECAE Routes

Route Name	Percent increase in cubic element surface area for different numbers of passes (N values)				
	0	1	2	4	8
A	0	41	103	235	502
B	0	41	67	158	345
C	0	41	0	0	0



Flow Plane of CuAg Composite

ECAE Tool Characteristics

	ARO	TEXAS
Billet Cross Section	19x19 mm	25x25 mm
Billet Length	200 mm	150 mm
Max Isothermal Temp.	500°C	300°C
Rapid Billet Ejection	Yes	Yes

ECAE Consolidation Processing Conditions

	Cu Nanopowder	Amorphous Zr-based
Encapsulation Material	Ni	Ni
Open or Closed Can	Vacuum	Vacuum
Extrusion Temp (°C)	23	400-440
Punch Speed (mm/s)	2.5	0.5
Max. Temp Rise (°C)	10	10-20
Max. Punch Load (kip/kN)	~190/~850	~80/~360
Est. Hydrostatic Pressure (ksi/MPa)	~160/~1100	~70/~480

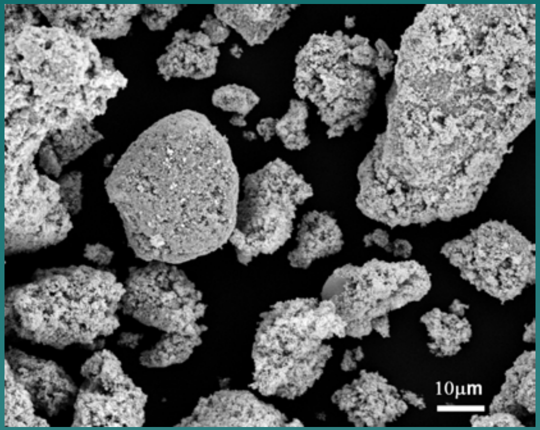
Cu Nanopowder Project Motivation

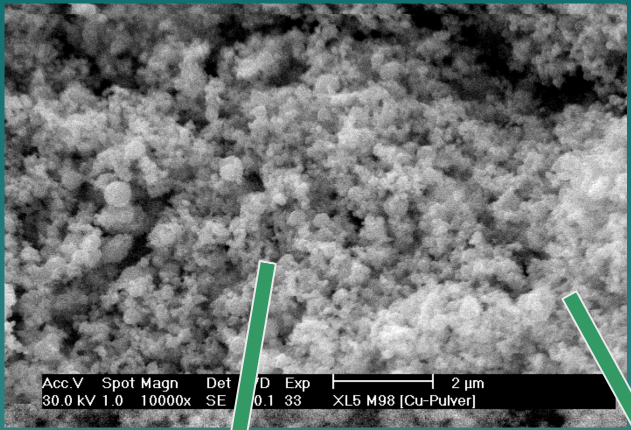
- Difficult to achieve grain sizes less than 100 nm using SPD techniques starting from coarse grain structures.
- Could consolidation of nanoparticles to full density be a method to obtain *bulk* samples?
- Investigate deformation and mechanical properties in *bulk* nanocrystalline materials.
- Conflicting results on fatigue response of UFG materials. Do the SPD microstructures really deteriorate the fatigue properties? Is it possible to improve ductility?

Note: Problems in nanoparticle consolidation: residual porosity, dynamic recrystallization, abnormal grain growth, bimodal porosity distribution, not much mechanical property data: only hardness measurements.

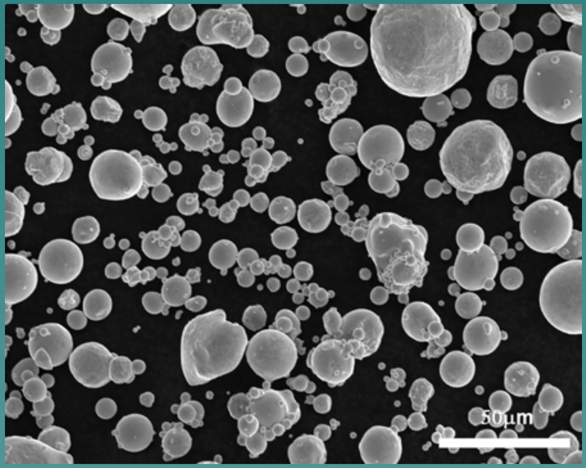
Initial Cu Powders

Electroexploded
Nanopowder
($O_2 \approx 0.1$ wt%)
(FNAA)

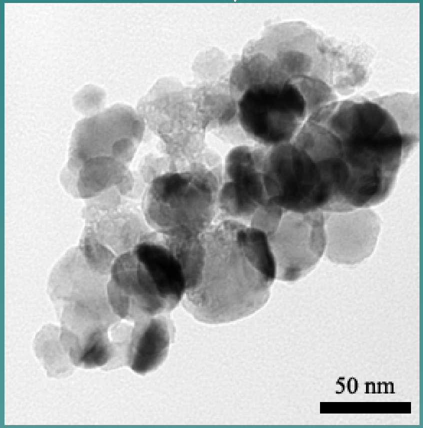




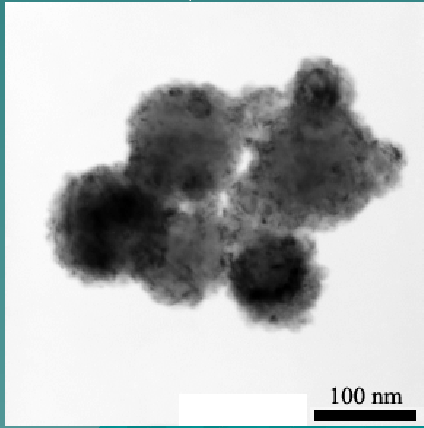
Agglomerates



Micropowder (DOE Ames)
99.99 wt% Cu, -325 mesh
Ave. Grain Size: 4.2 microns (X-Ray analysis)



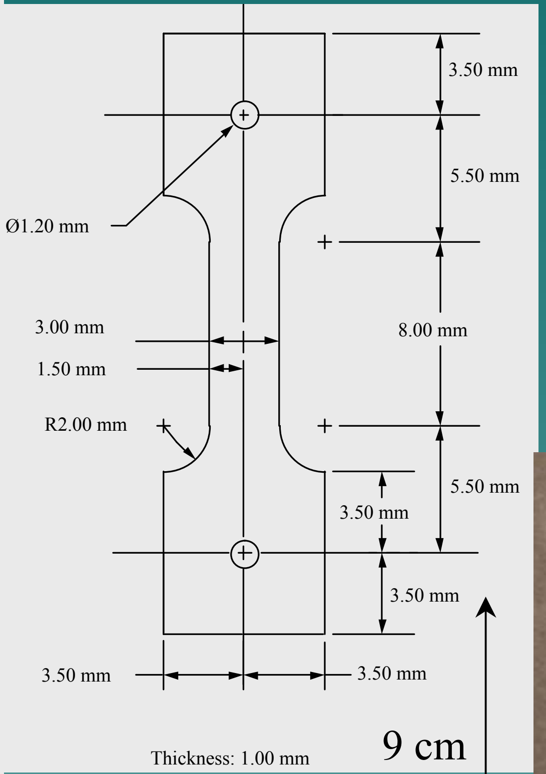
Average size 67 nm
(X-Ray analysis)

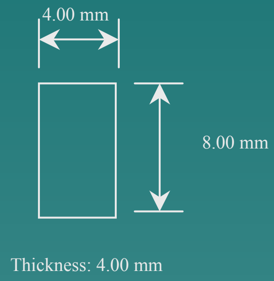


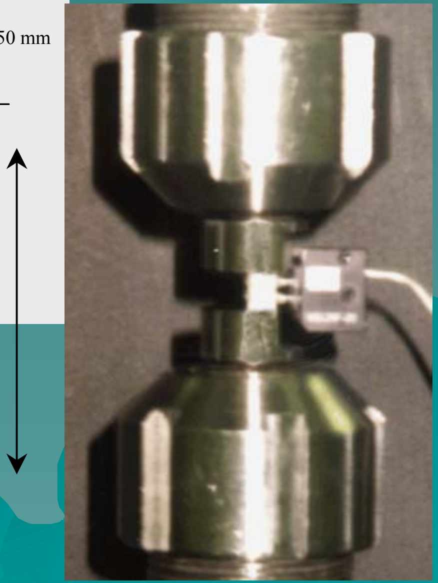
Average size 130 nm
(X-Ray analysis)

Extrusion and Testing Conditions

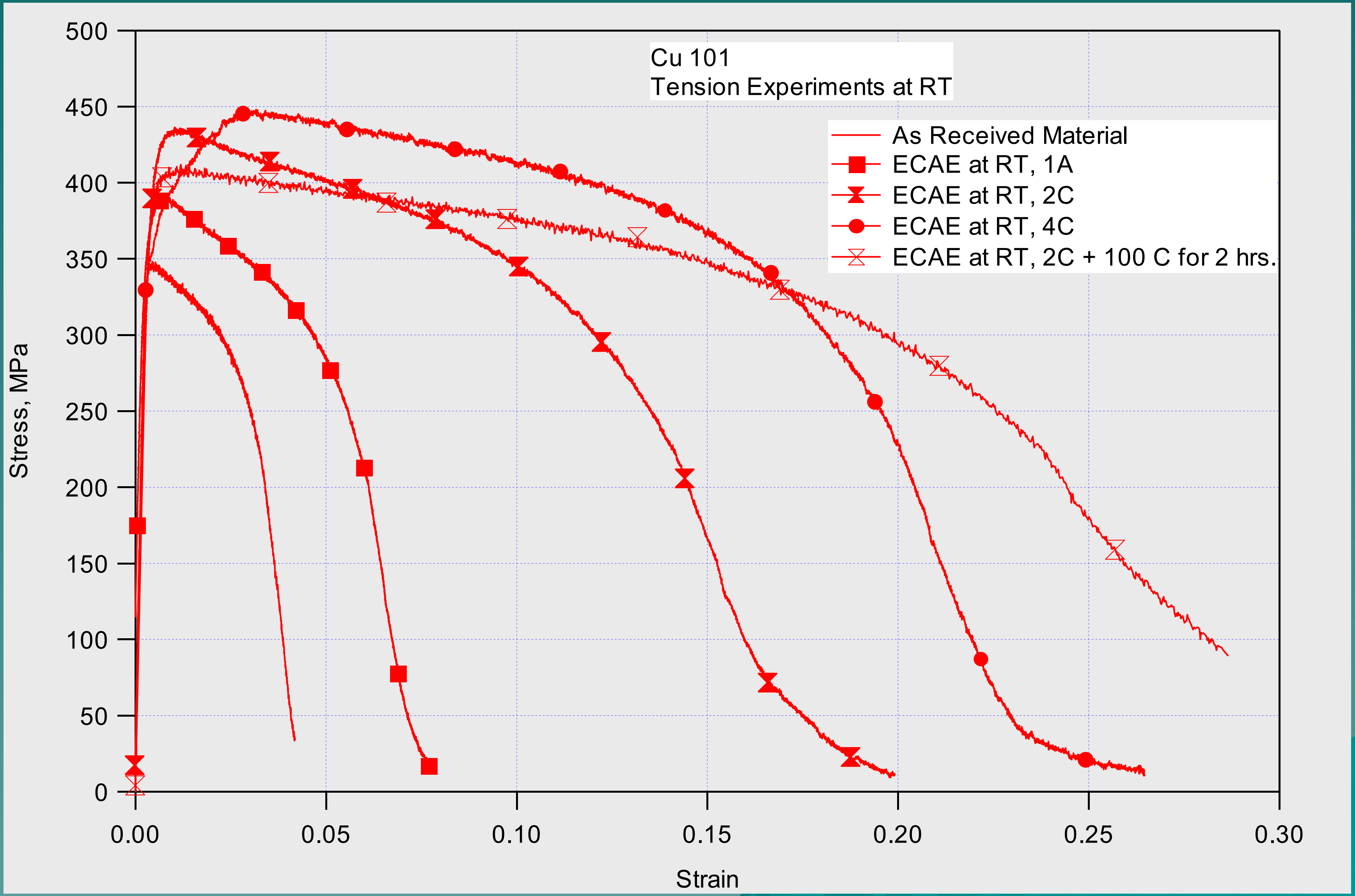
ECAE Route	Can Material	Extrusion Speed	Powder Size
1A	Copper	0.1"/sec	- 325 mesh
2A			
2B			
2C			
4C			
2B	Nickel	0.1"/sec	130 nm
2C			
4E			
Annealed Bulk Copper			75 μm



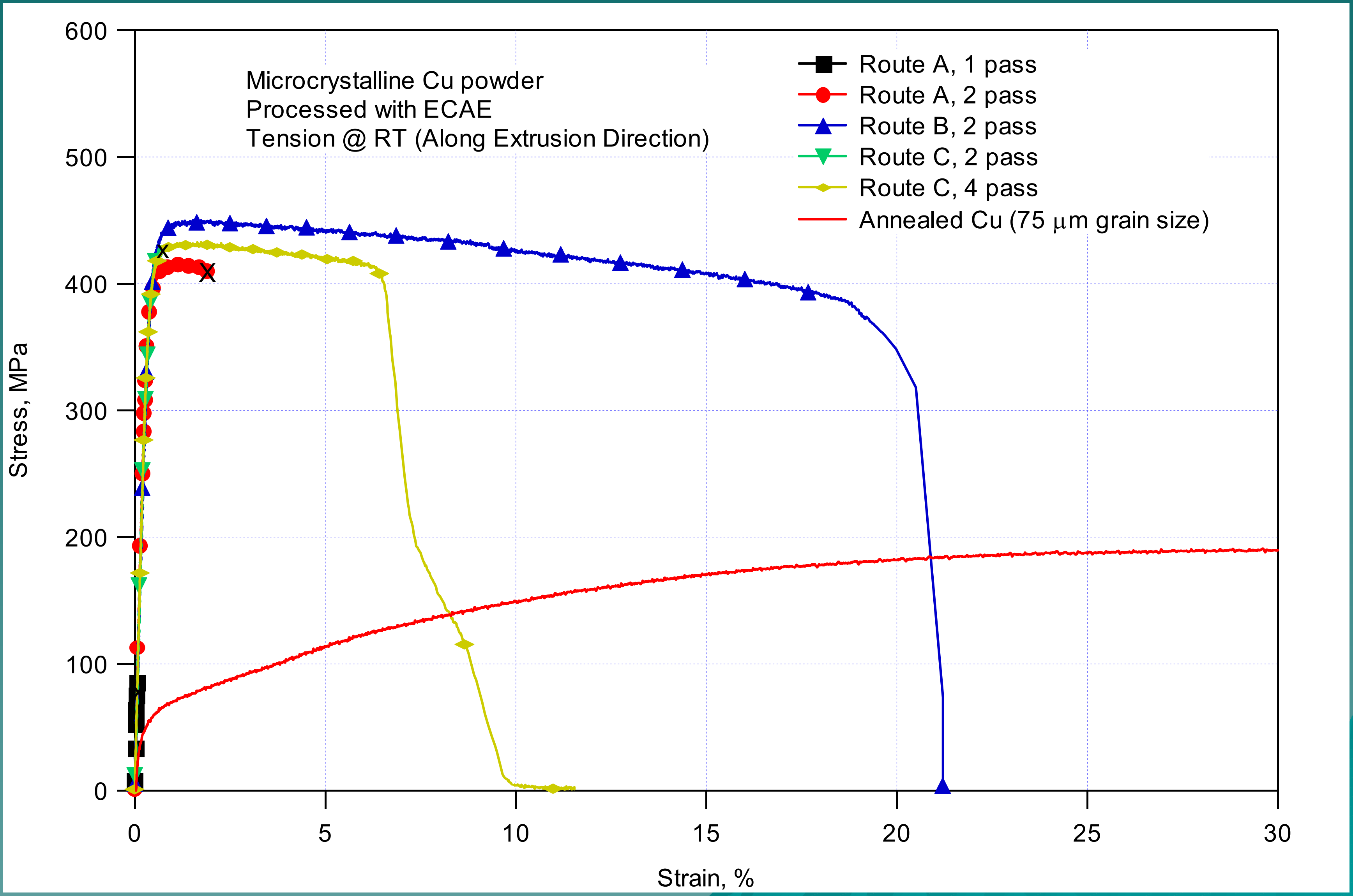




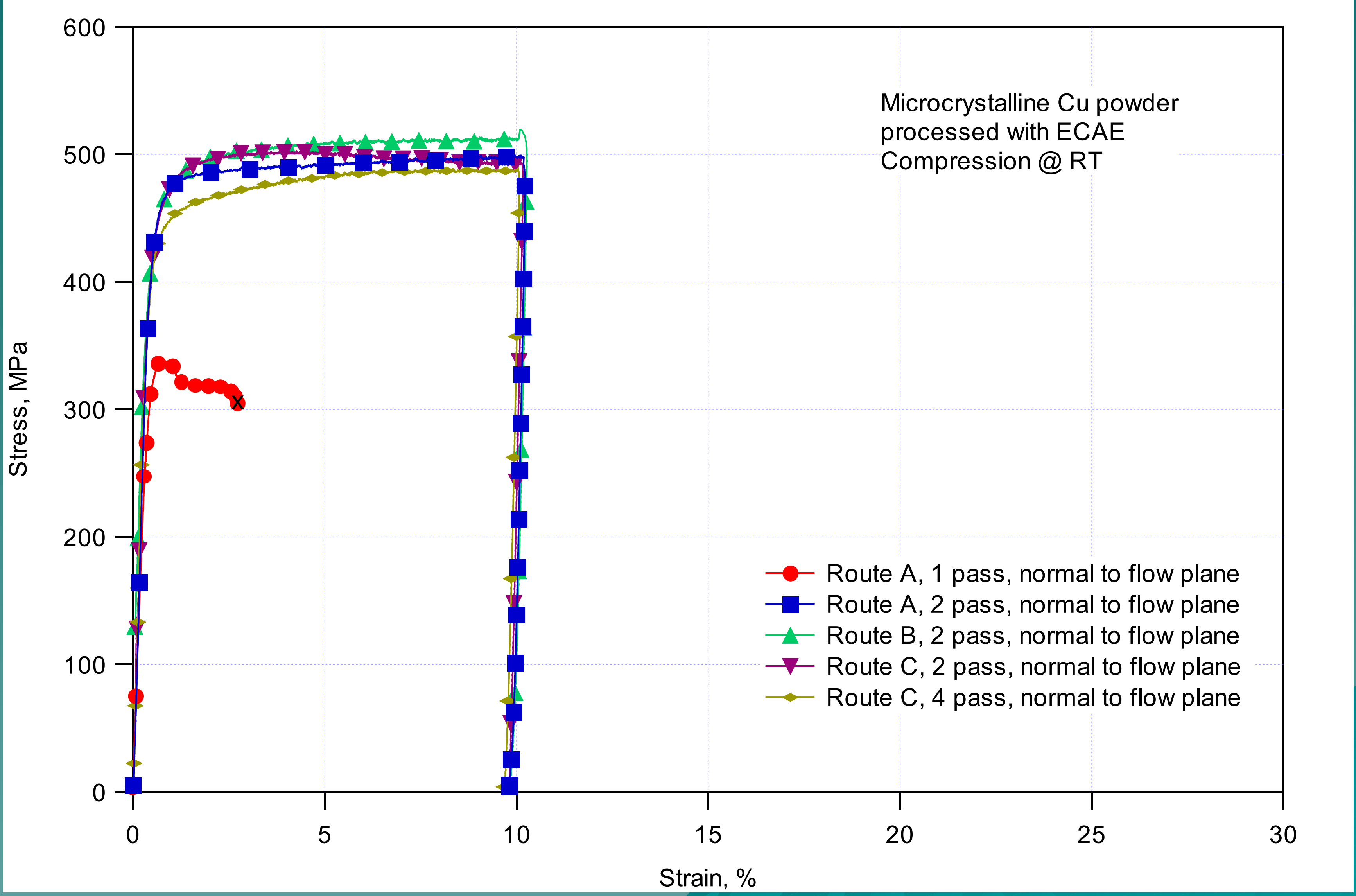
Tension Experiments
ECAE Processed Bulk Samples



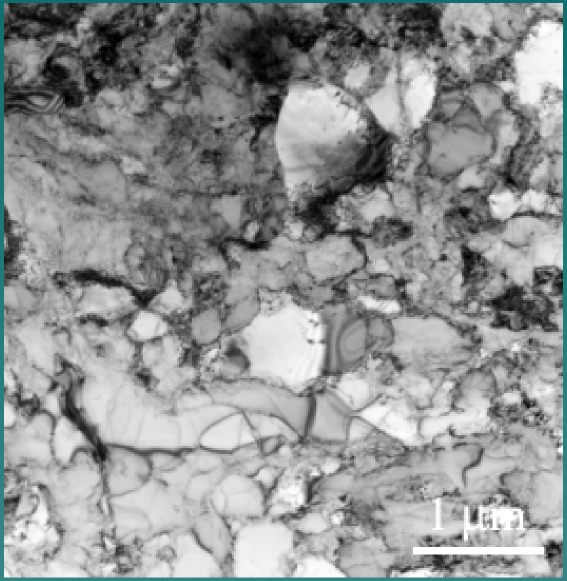
Microcrystalline Powder Consolidate Tension Experiments



Microcrystalline Powder Consolidate Compression Experiments



Microstructural Evolution of Microcrystalline Powder Consolidate

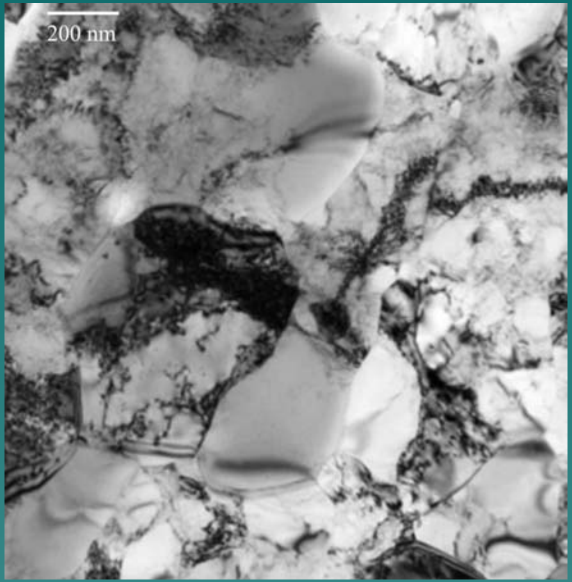


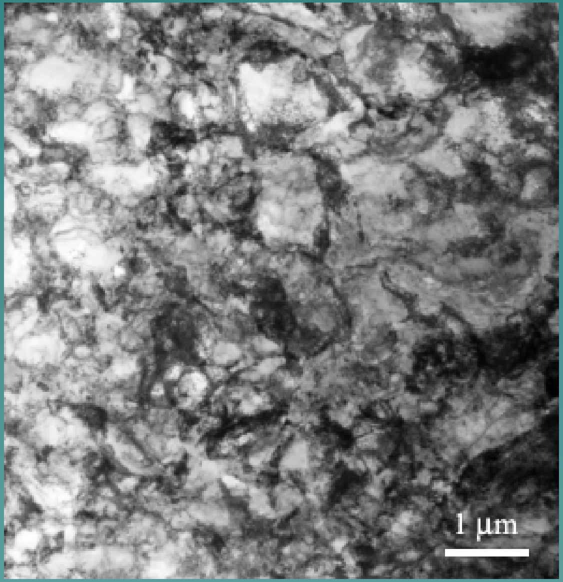
2A

both high and low dislocation density areas

2B

~ 200 nm dislocation free subgrains



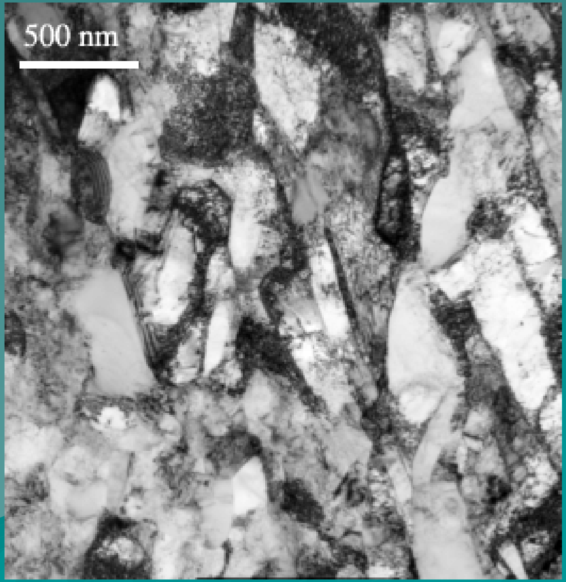


2C

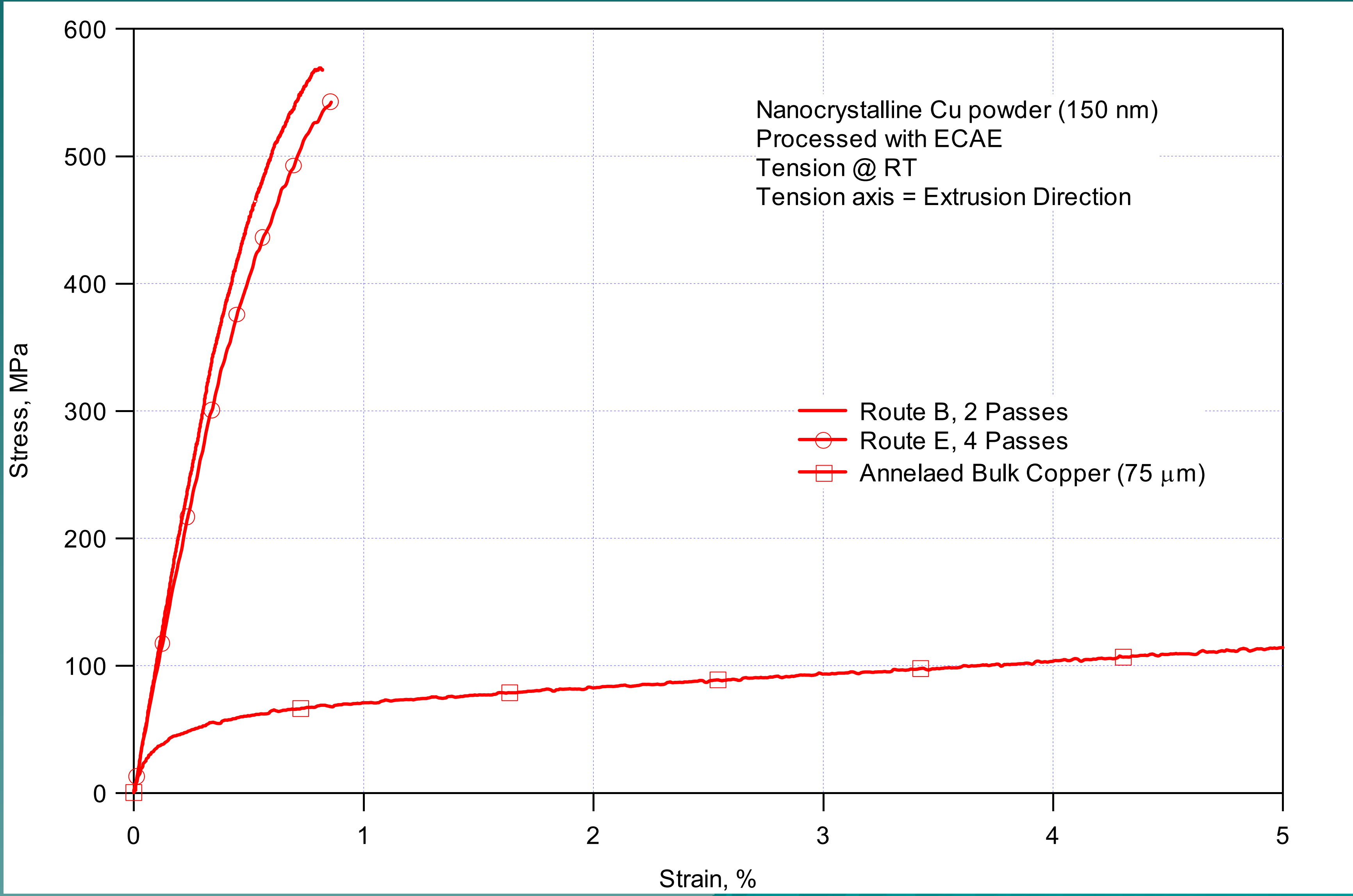
very high dislocation density, not well-developed subgrains

4C

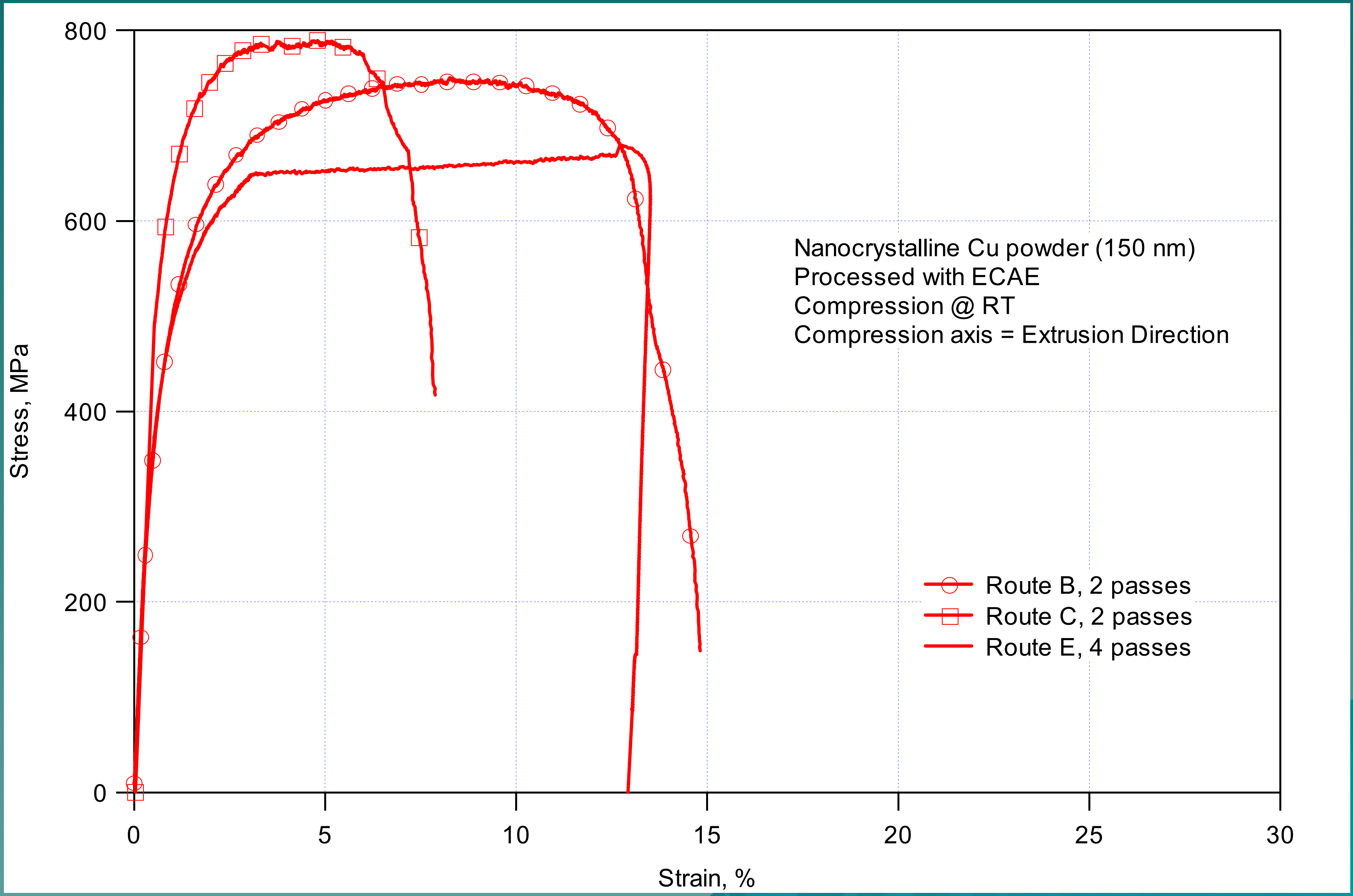
very high dislocation density, well-developed subgrains



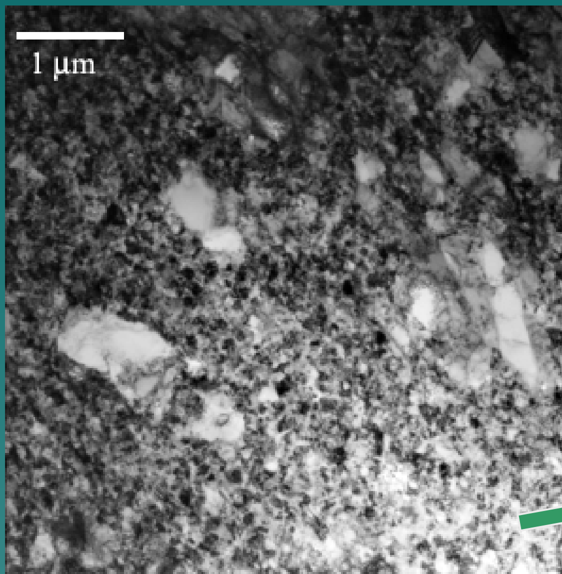
Nanocrystalline Powder Consolidate Tension Experiments



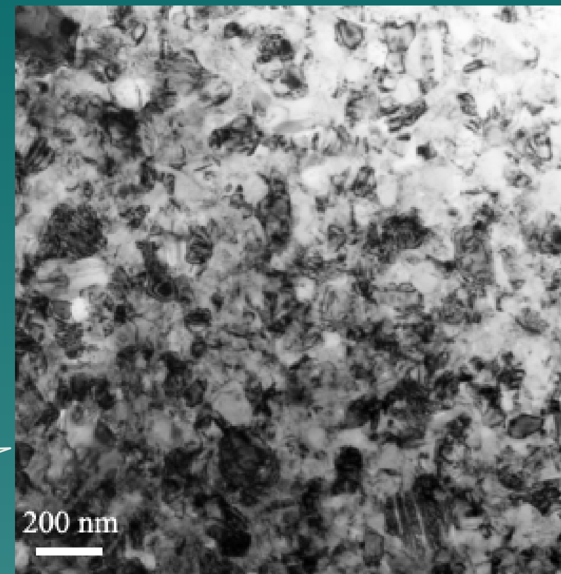
Nanocrystalline Powder Consolidate Compression Experiments

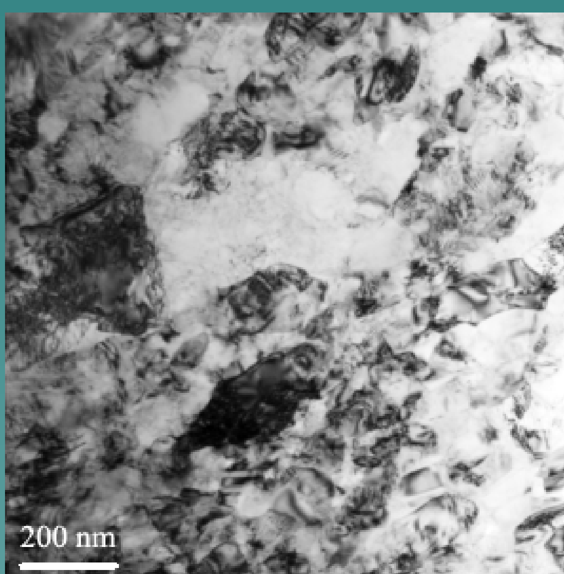


Microstructural Evolution of Nanocrystalline Powder Consolidate

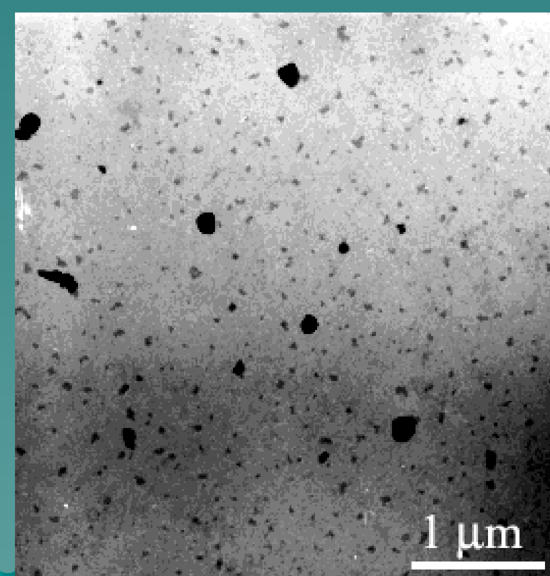


2C



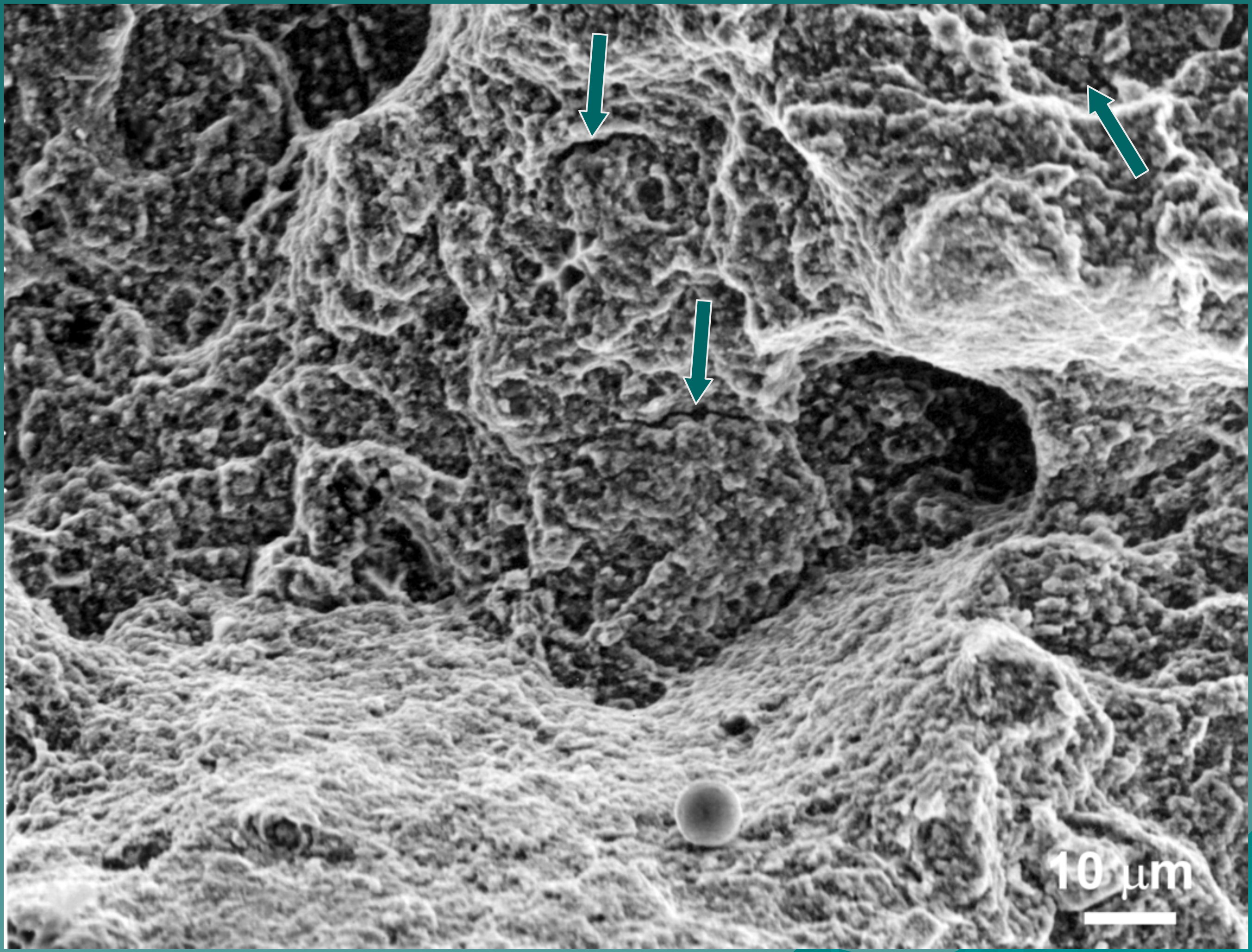


2B



Initial Powder

Fracture Surface of Nanocrystalline Powder Consolidate



Grain Size vs. Strength Relationship

ECAE Route	Powder Size	Grain Size		Tension (Extrusion Direction)				Compression		
		X-Ray	TEM	E (GPa)	σ_y (0.2%)	σ_{UTS}	ε_f (%)	E (GPa)	σ_y (0.2%)	σ_{UTS}
1A	- 325 mesh (4.2 μm from X-Ray)	-	-	109	-	-	-	81	330 (FD)	335 (FD)
2A		315 nm	200 – 300 nm (some grains >500 nm)	115	406	420	1.9	110	437 (FD)	477(FD)
2B		300 nm	200 – 300 nm (some grains < 100 nm)	108	433	470	19.2	93	428 (FD)	488 (FD)
2C		250 nm	200 - 300 nm	114	-	430	0.5	106	440 (FD)	489 (FD)
4C		260 nm	250 nm	115	418	443	9.4	111	418 (FD)	460 (FD)
2B	130 nm (from X-Ray, about 100 nm from TEM)	110 nm	70 - 100 nm	104	559	573	0.81	101	399 (ED)	694 (ED)
2C		140 nm	~200 nm and 50 - 80 nm	-	-	-	-	92	560 (ED)	760 (ED)
4E		-	~250 nm and 40 – 80 nm	92	516	546	0.7	90	473 (ED)	628 (ED)
ECAE processed bulk Cu (1A)		-	200nm- (>1 μm in the elongated direction)	120	287	395	7.4	-	-	-
ECAE processed bulk Cu (2C)		-	200-500nm	116	310	441	18.2	-	-	-
ECAE processed bulk Cu (4C)		-	200-500nm	125	346	463	23.4	-	-	-
Annealed Bulk Copper		-	75 μm	120	51	256	37.2	-	-	-

FD = Flow Direction, ED = Extrusion Direction

Conclusions

- Successful consolidation of microcrystalline copper particles to full density. Route 2B resulted in the best results.

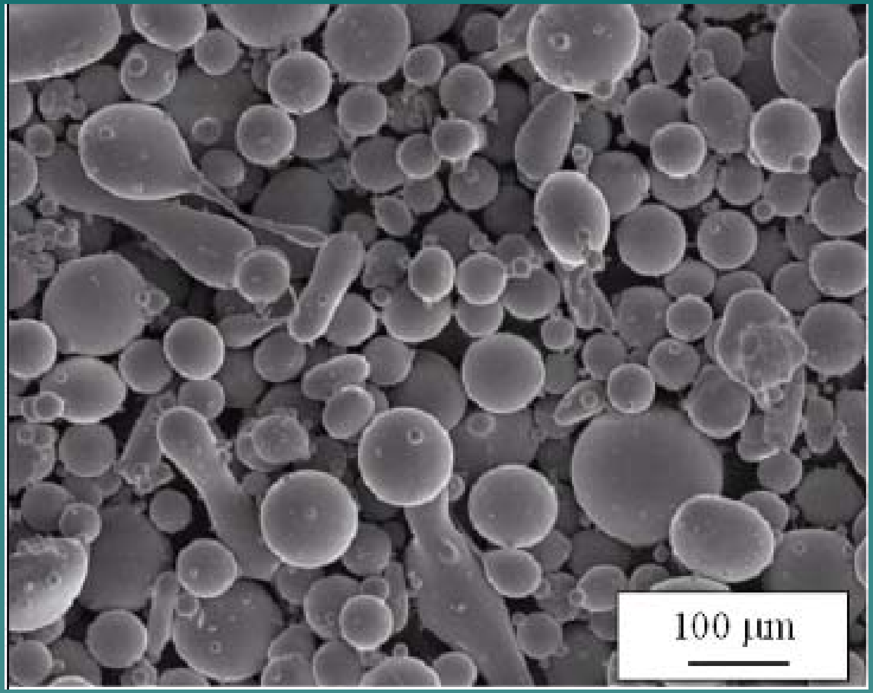
- Nanoparticles were consolidated with relative success. Tensile strength of 550 MPa and Compressive strength of 780 MPa were achieved. Low tensile ductility is attributed to the inter-agglomerate debonding which might be due to moisture or an oxide layer.

- ECAE appears to be a viable approach to obtain bulk nanocrystalline (<100 nm) materials for structural applications.

Zr-based Metallic Glass Powder Consolidation Motivation / Approach

- Interest in production of bulk amorphous metal for structural applications.
- Approach:
  - ECAE consolidation of gas-atomized Zr-based amorphous metal powder into bulk amorphous metal. Vitreloy 106a is chosen because of a large T_x-T_g.
  - Compostion: ($Zr_{58.5}Nb_{2.8}Cu_{15.6}Ni_{12.8}Al_{10.3}$)
- Objectives:
  - No crystallization
  - Good particle-to-particle bonding
  - Tensile strength comparable to cast counterpart
  - Consolidate dimensions greater than casting

Initial Amorphous Zr-based Powders



As-Received Powder

- **Powder characteristics**
  - **$Zr_{58.5}Nb_{2.8}Cu_{15.6}Ni_{12.8}Al_{10.3}$**
  - **Gas atomized at AMES-MPC**
  - **38 µm < Diameter < 150µm**
  - **Batch 1: ~ 1280 ppmw oxygen (0.57 at %) and ~ 266 carbon ppmw in dia. < 75 µm**
  - **Batch 2: ~ 780 ppmw oxygen**
  - **Amorphous character**

Batch 1: T_g = 398 °C; T_x = 460 °C

ΔT = 62 °C

Batch 2: T_g = 403 °C; T_x = 480 °C

ΔT = 77 °C

Extrusions Conditions

Billet ID	Cu050	Cu051	Cu052	Cu053	Cu054	Cu058	Ni024	Ni023	Ni029	Ni029	Ni041
Extrusion Route	1A	1A	1A	1A	1A	1A	1A	1A	1A	2B	2C
T_{die} (°C)	440	420	420	420	400	430	410	430	410	420	410/420
$T_{maximum}$ (°C)	459	NA	NA	NA	420	451	415	433	421	NA	NA
Punch Speed (mm/s)	6	1	6	12	6	6	0.5	0.5	0.5	0.5	0.5
Time above T_g (s)	NA	NA	NA	NA	NA	148	195	382	231	NA	NA
Time above T_{tool} (s)	NA	NA	NA	NA	NA	23	77	44	147	NA	NA
Microhardness (HV_{500})	520 ± 35	493 ± 25	475 ± 15	480 ± 20	480 ± 33	484 ± 15	488 ± 4	490 ± 5	NA	493 ± 5	497 ± 4
	Batch 1 (1280 ppmw O_2) (HV_{500} = 467 ± 35)						Batch 2 (780 ppmw O_2) (HV_{500} = NA)				

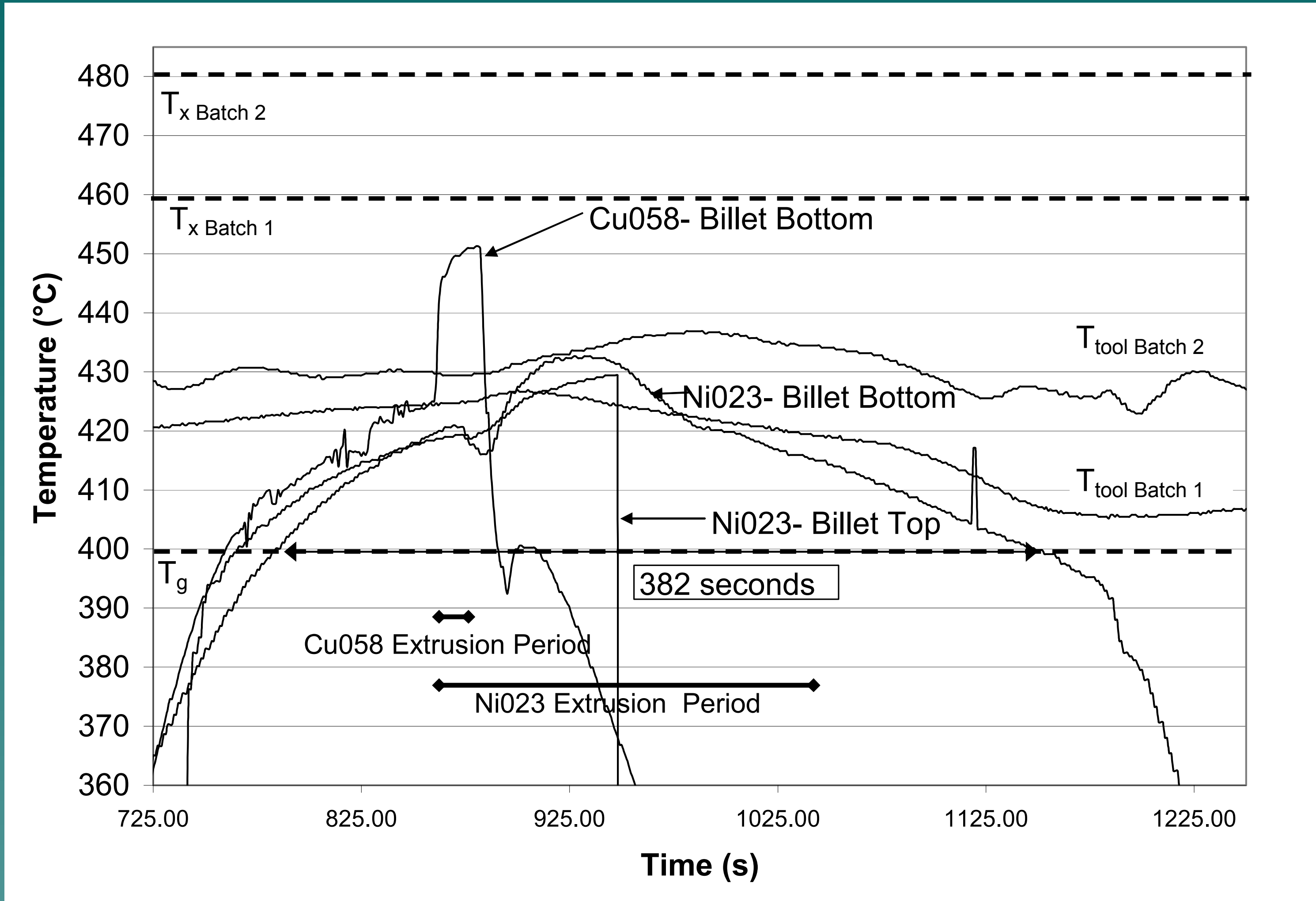
- Nickel cans contain V106a with 780 ppmw oxygen (T_g = 403°C - T_x=480 °C)

- Copper cans contain V106a with 1280 ppmw oxygen (T_g = 398°C - T_x=460 °C)

Controlled Variables:

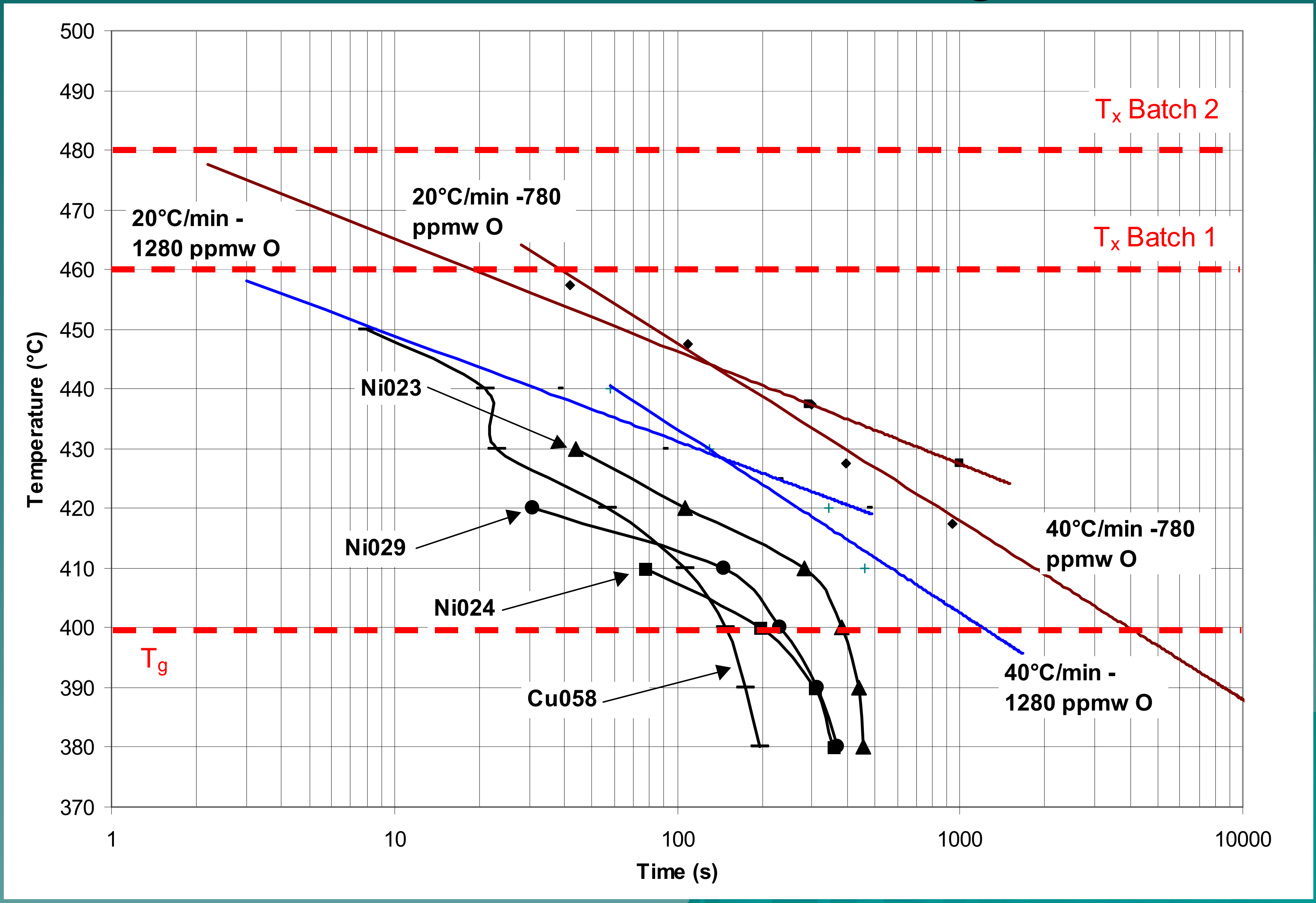
- Temperature
- Strain Rate
- Extrusion Rate
- Oxygen Content
- Hydrostatic Pressure

Billet and Die Temperature during ECAE

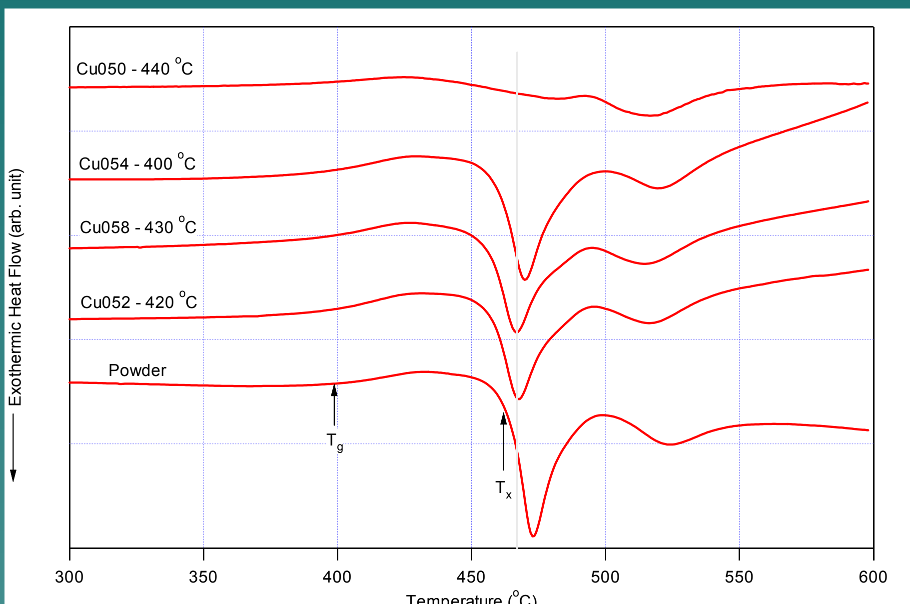


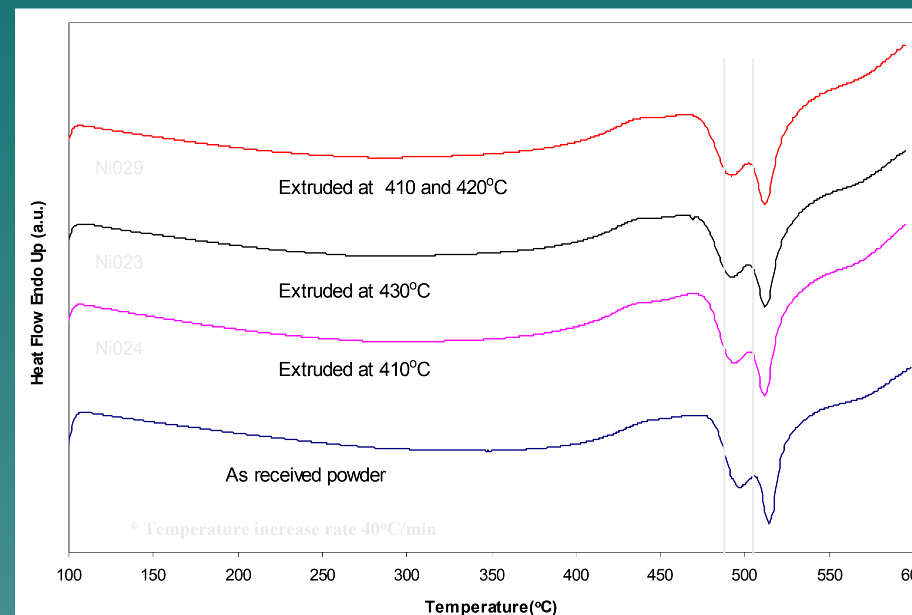
Temperature versus time for billets Cu058 and Ni023 showing the sample time above Tg and the rise in temperature due to material deformation as it passes through the shear zone. Both billets were extruded with the die at 430°C and with a punch speed of 6 mm/s and 0.5 mm/s for Cu058 and Ni023, respectively. Note that the horizontal axis is only a time scale and not an indicator of the amount of time into the processing.

Thermal History of V106a Powder Consolidations on TTT Diagram



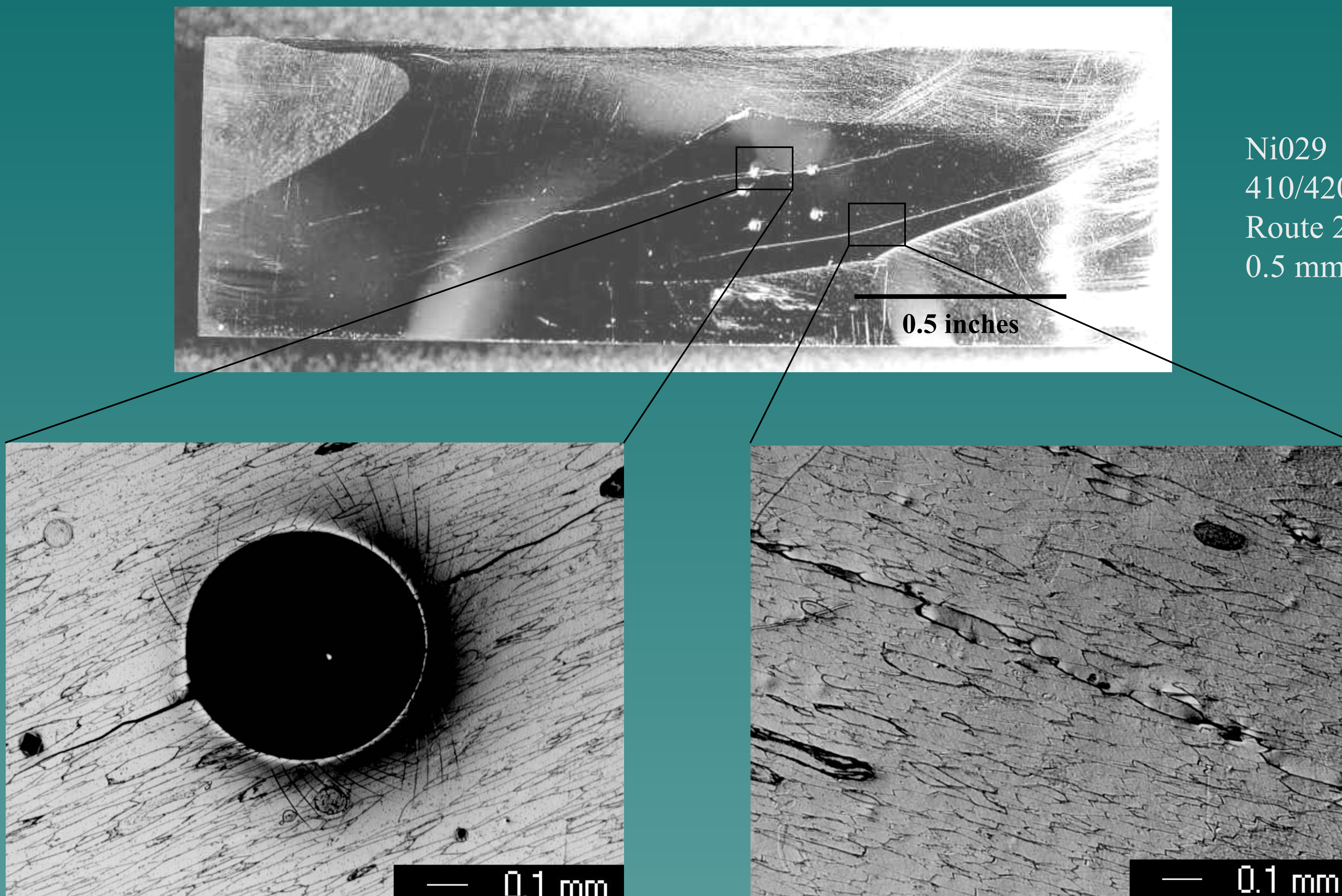
DSC Curves for V106a Powder and Consolidates





- Combined effects of oxygen content, temperature, strain rate, and can material

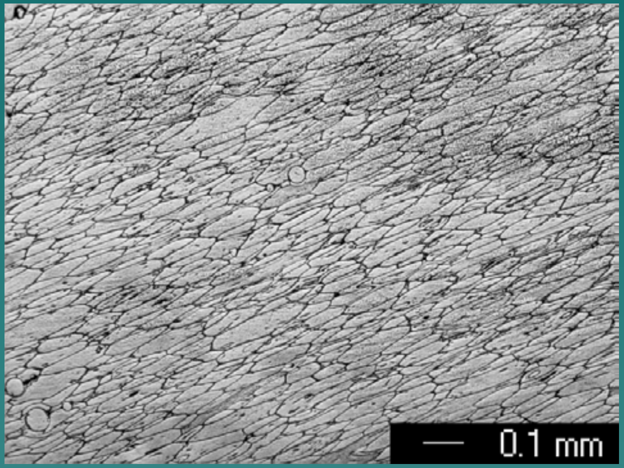
Fine Interparticle Cracking



Ni029
410/420 ^{o}C
Route 2B
0.5 mm/s

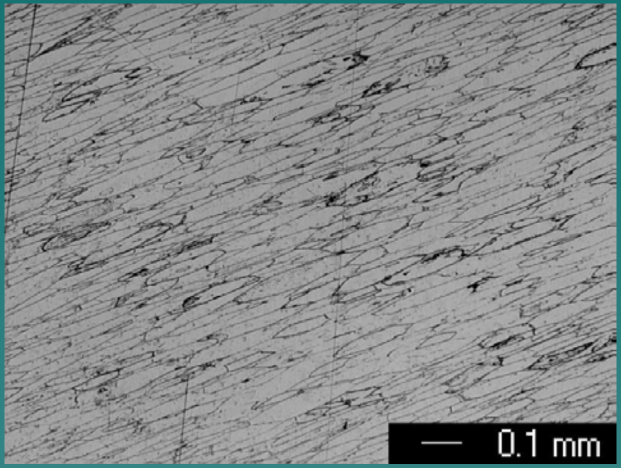
Effect of Oxygen Content

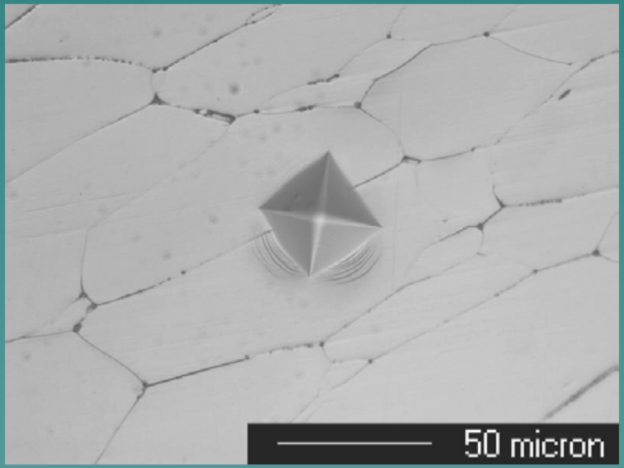
1280 ppmw			780 ppmw



Cu058
430 °C
Route 1A
6 mm/s

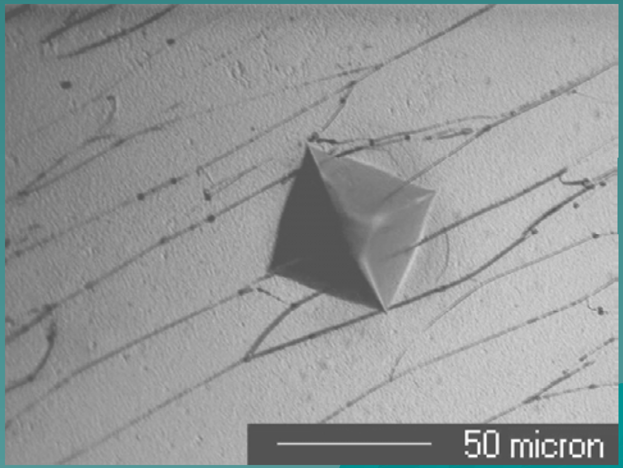
Ni023
430 °C
Route 1A
0.5 mm/s



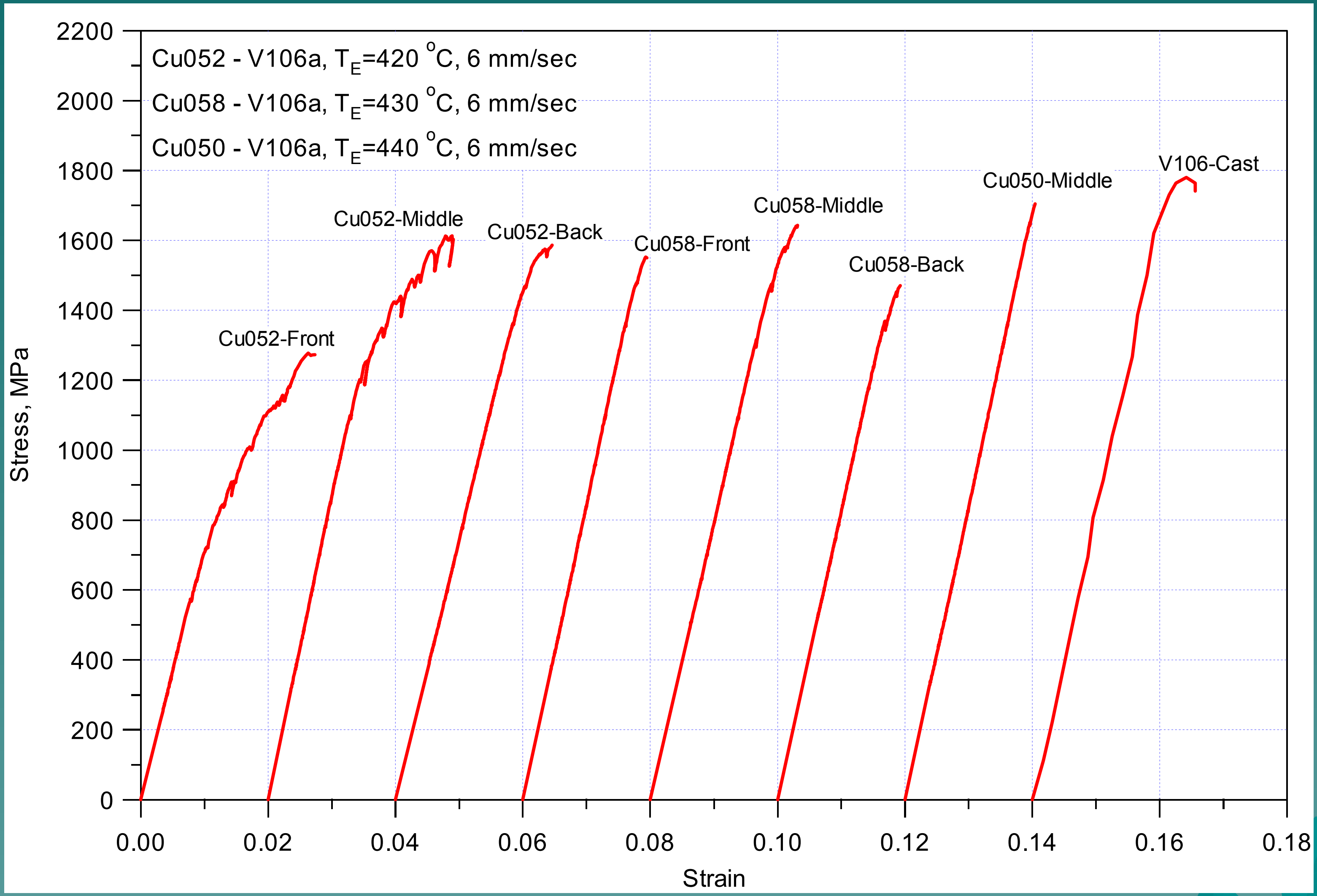


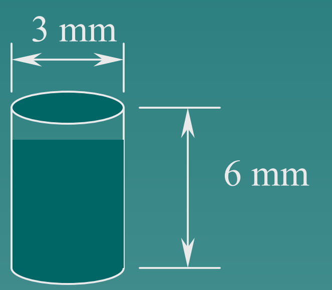
Cu052
420 °C
Route 1A
6 mm/s

Ni029
410/420 °C
Route 2B
0.5 mm/s



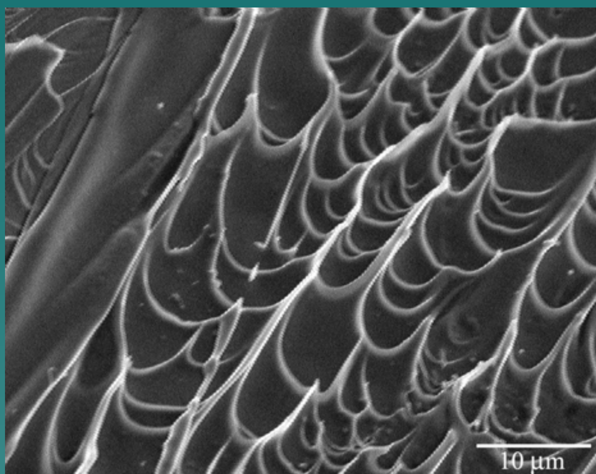
Compressive Response of Consolidated V106a Powder

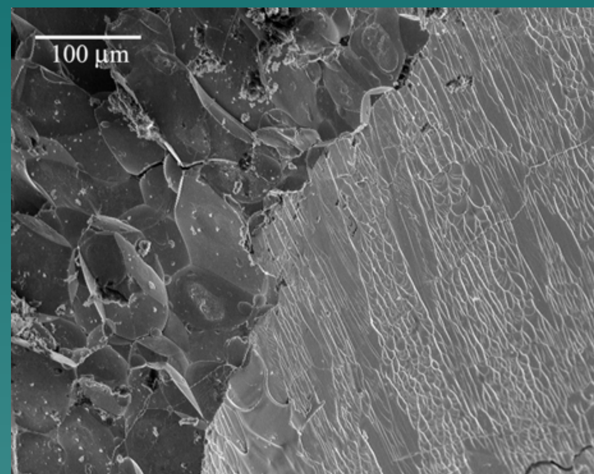


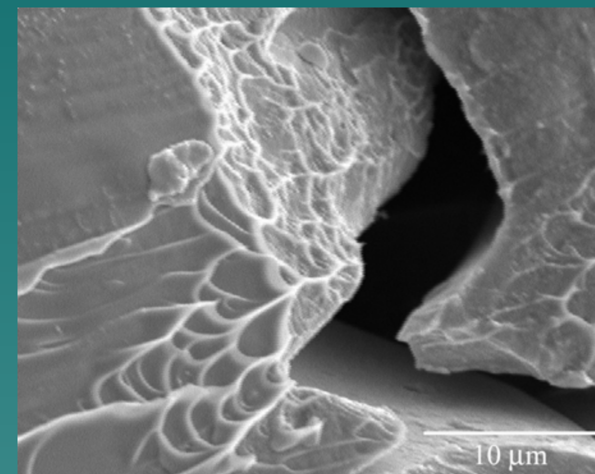


V106 compression curve is taken from Choi-Yim et al., Acta Mater., 2002.

Compressive Fracture Surfaces







430 ^{o}C, 6 mm/sec

420 ^{o}C, 6 mm/sec

Conclusions

- Full consolidation with one ECAE pass at temperatures of T_g and higher without significant crystallization.
- Fine inter-particle cracks are sometimes present in the consolidate
- Higher oxygen content level restricts t-T space for consolidation, decreases ductility, decreases ΔT, promotes crystallization and inhibits interparticle bonding due to surface oxides.
- Zr-based amorphous metal powders with a substantial TTT opportunity window can be consolidated in the supercooled liquid region by ECAE

Lessons Learned

- Problems Identified
  1. It is difficult to achieve grain sizes ≤ 100nm using ECAE when starting from coarse grain structures.
  2. **Residual porosity** (from initial powder agglomerates) may be a problem for nanoparticle consolidation and mechanical properties.
  3. **Particle surface contamination** (Oxygen, water, etc.) is highly detrimental to effective consolidation.
  4. Severe plastic deformation may result in **local heating** and dynamic recrystallization (for crystalline phases) or crystallization (for amorphous phases).
  5. Brittle material (whether the precursor is particulate or bulk) is difficult to ECAE process without **cracking**.

Lessons Learned

- Encouraging Results

1. Cu nanopowder can be effectively consolidated by ECAE: One pass gives nearly full density; two passes improves mechanical properties.

2. ECAE consolidated Cu nanopowder has higher tensile and compression strengths than does wrought Cu given severe plastic deformation.

3. Amorphous metal powder, with a substantial supercooled liquid region, can be consolidated to nearly full density by one pass ECAE without significant crystallization

Questions Remain

1. Are the properties of ECAE processed powder consolidates isotropic?
2. How do HIPing, area reduction extrusion and ECAE compare with respect to effectiveness (level of material properties in consolidate and ease of processing) of powder consolidation?
3. Can the ECAE process for powder consolidation be scaled up for the production of high efficiency structural components?
4. Can amorphous metal powder be consolidated below T_g?